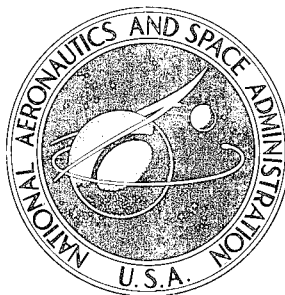
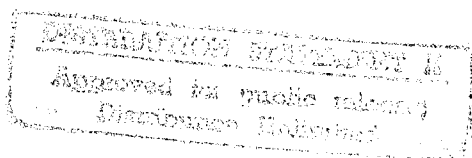


NASA TECHNICAL NOTE



NASA TN D-6714

NASA TN D-6714



19960607 154

GRAPHITE FLUORIDE  
AS A SOLID LUBRICANT  
IN A POLYIMIDE BINDER

*by Robert L. Fusaro and Harold E. Sliney*

*Lewis Research Center*

*Cleveland, Ohio 44135*

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION • WASHINGTON, D. C. • MARCH 1972

REPRODUCTION UNLIMITED

19960607 154

1. Report No. NASA TN D-6714	2. Government Accession No.	3. Recipient's Catalog No.	
4. Title and Subtitle GRAPHITE FLUORIDE AS A SOLID LUBRICANT IN A POLYIMIDE BINDER		5. Report Date March 1972	
		6. Performing Organization Code	
7. Author(s) Robert L. Fusaro and Harold E. Sliney		8. Performing Organization Report No. E-6694	
9. Performing Organization Name and Address Lewis Research Center National Aeronautics and Space Administration Cleveland, Ohio 44135		10. Work Unit No. 132-15	
		11. Contract or Grant No.	
12. Sponsoring Agency Name and Address National Aeronautics and Space Administration Washington, D.C. 20546		13. Type of Report and Period Covered Technical Note	
		14. Sponsoring Agency Code	
15. Supplementary Notes			
16. Abstract Polyimide resin (PI) was shown to be a suitable binder material for the solid lubricant graphite fluoride ((CF <sub>1.1</sub> ) <sub>n</sub> ). Comparisons were made to similar tests using PI-bonded MoS <sub>2</sub> films, (CF <sub>1.1</sub> ) <sub>n</sub> rubbed films, and MoS <sub>2</sub> rubbed films. The results showed that, at any one specific temperature between 25° and 400° C, the wear life of PI-bonded (CF <sub>1.1</sub> ) <sub>n</sub> films exceeded those of the other three films by at least a factor of 2 and by as much as a factor of 60. Minimum friction coefficients for the PI-bonded films were 0.08 for (CF <sub>1.1</sub> ) <sub>n</sub> and 0.04 for MoS <sub>2</sub> . The rider wear rates for the two PI-bonded films at 25° C were nearly equal.			
17. Key Words (Suggested by Author(s)) Solid lubricant    High-temperature lubrication Graphite fluoride    Bonded coating Polyimide		18. Distribution Statement Unclassified - unlimited	
19. Security Classif. (of this report) Unclassified	20. Security Classif. (of this page) Unclassified	21. No. of Pages 17	22. Price* \$3.00

# GRAPHITE FLUORIDE AS A SOLID LUBRICANT IN A POLYIMIDE BINDER

by Robert L. Fusaro and Harold E. Sliney

Lewis Research Center

## SUMMARY

A solid lubricant film, consisting of a polyimide resin (PI) as the binder and graphite fluoride ( $(\text{CF}_{1.1})_n$ ) as the lubricant, was formulated. The lubricant was applied to roughened 440C stainless-steel disks ( $0.90 \times 10^{-6}$  to  $1.25 \times 10^{-6}$  m, rms) using an artist's airbrush. The thicknesses of the films were in the range of 10 to 20 micrometers. A pin-on-disk friction apparatus was used to evaluate the friction coefficient, the wear life, and the rider wear rate. The test conditions were a dry-air atmosphere (moisture content, 20 ppm), 440C stainless-steel riders, a 1-kilogram load, and a 2.6-meter-per-second (1000-rpm) sliding speed.

For comparison, similar experiments were conducted using PI-bonded molybdenum disulfide ( $\text{MoS}_2$ ) films,  $(\text{CF}_{1.1})_n$  rubbed films, and  $\text{MoS}_2$  rubbed films. The results indicated that PI is a suitable binder material for the solid lubricant  $(\text{CF}_{1.1})_n$ . Comparison of the wear lives at any one specific temperature between  $25^\circ$  and  $400^\circ$  C showed that the PI-bonded  $(\text{CF}_{1.1})_n$  films were at least two times better than the other three films at  $25^\circ$  C and as much as 60 times better than either  $\text{MoS}_2$  film at  $400^\circ$  C.

The minimum friction coefficient for the PI-bonded  $(\text{CF}_{1.1})_n$  film was 0.08 over the entire test temperature range of  $25^\circ$  to  $500^\circ$  C. The minimum friction coefficient for the PI-bonded  $\text{MoS}_2$  film was 0.04 over the range of  $25^\circ$  to  $300^\circ$  C. Above this temperature, the friction coefficient increased gradually until, at  $450^\circ$  C, no minimum value was recorded (i. e., the film failed immediately).

The rider wear rates of the two PI-bonded films at  $25^\circ$  C were nearly equivalent. These wear rates were, however, only one-tenth the wear rate of the rubbed-on films. The upper temperature limit, in air, for the PI-bonded films was deemed to be  $400^\circ$  C, since at this temperature the coatings became powdery.

## INTRODUCTION

In a previous report (ref. 1), it has been shown that burnished (rubbed-on) films of graphite fluoride ( $(\text{CF}_{1.1})_n$ ) have excellent lubricating properties. The experiments

described in reference 1 consisted of rubbing  $(CF_{1.1})_n$  powders onto roughened 440C stainless-steel disks and then evaluating the resulting films with a pin-on-disk friction apparatus (fig. 1). The results indicated that the minimum value of friction coefficient for these  $(CF_{1.1})_n$  lubricated disks was comparable to, or better than, the friction coefficients for disks similarly lubricated with molybdenum disulfide ( $MoS_2$ ) or graphite. The wear life of the rubbed  $(CF_{1.1})_n$  films was as much as six times better than that of either of the two other lubricant films.

Solid lubricants, such as  $MoS_2$  or graphite, usually show increased wear life when they are incorporated into a binder system. The object of this investigation was, therefore, to determine whether  $(CF_{1.1})_n$  would also have improved wear life when used with

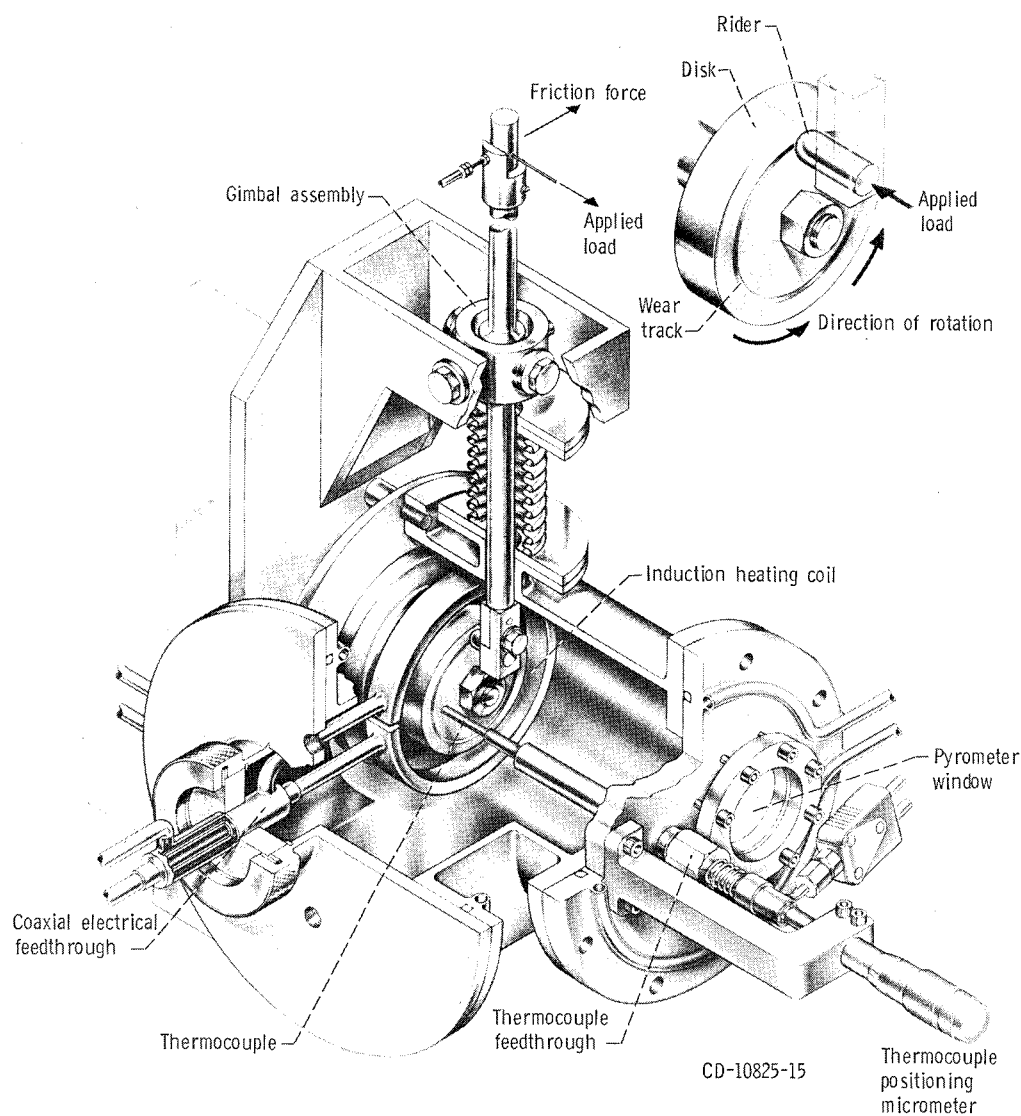


Figure 1. - Friction and wear testing device.

a binder material. A secondary objective was to compare the results to those obtained when  $\text{MoS}_2$  was used with the same binder material.

The binder material used in these experiments was a polyimide resin (manufacturer's designation, PI-4701) (ref. 2). Polyimide has been shown in previous Lewis Research Center reports (refs. 3 to 6) to be a good friction and wear material. Also reference 7 has reported good results using it as a binder material for  $\text{MoS}_2$ . In addition,  $(\text{CF}_{1.1})_n$  was found to mix readily with the polyimide varnish. For these reasons and because it was stable in air to over  $400^\circ\text{C}$ , it was chosen as the binder material.

The experimental conditions used in these tests were a 2.6-meter-per-second (1000-rpm) sliding speed, a 1-kilogram load, a dry-air atmosphere (moisture content, 20 ppm), and 440C stainless-steel riders and disks.

## MATERIALS

### Graphite Fluoride

A brief review of the history and physical properties of graphite fluoride  $((\text{CF}_{1.1})_n)$  is given in reference 1. After the publication of that work, the existence of a patent entitled "Dry Lubrication" (ref. 8) was discovered. The patent was granted on July 25, 1961; it describes a product with a carbon-to-fluorine ratio of 1:1. The patent states, "Fluorinated graphite has outstanding dry lubricating properties." However, no data supporting this statement were included with the patent and quantitative information was not available prior to reference 1.

More recently, Ishikawa and Shimada (ref. 9) have used graphite fluoride as an additive in grease, mechanical carbons, and polytetrafluoroethylene (PTFE) - fibrous carbon composites. In each instance, beneficial results were reported. They found increased load-carrying capacity, reduced surface temperatures, or seizure prevention.

Gisser, Petronio, and Shapiro (ref. 10) have also conducted some exploratory tests on the friction and wear properties of graphite fluoride. They found that, by adding 2-percent graphite fluoride to a lithium soap - diester grease, low friction could be attained at temperatures up to  $344^\circ\text{C}$ . The grease alone or with graphite added failed at  $215^\circ\text{C}$ . They also tested graphite fluoride in a silicate binder and in an epoxy-phenolic binder. In general, they found that films formulated with graphite fluoride gave better friction and wear results than similar films formulated with graphite.

### Polyimides

Most polymers lose their rigidity and strength at elevated temperatures (refs. 11

and 12). One of the reasons for this is that their chain-like structure is weakened by the increased molecular vibrations associated with increased temperatures. There are two possible consequences of these vibrations: Either the bonds between the monomer units break and the polymer disintegrates, or the polymer melts.

To obtain polymers with improved thermal stability, it is necessary to devise a means of stiffening the molecules so that heat and its resulting vibrations will have less effect (ref. 12). A method of accomplishing this is by the insertion of aromatic rings (rings of carbon atoms) into the polymer chains (refs. 13 to 15). These resulting polymers are designated aromatic polymers. Polymers of this type have an upper temperature limit of about 300° C (refs. 12 and 15).

To make polymers which will withstand temperatures above 300° C, the parent molecule itself is made into a ring. These rings (called heterocyclic molecules) are then alternated with the aromatic rings to form cyclic-chain polymers. One class of these polymers has been designated aromatic polyimides (refs. 14 to 20). Their basic structure is shown in figure 2, where R represents a thermally stable group (refs. 14, and

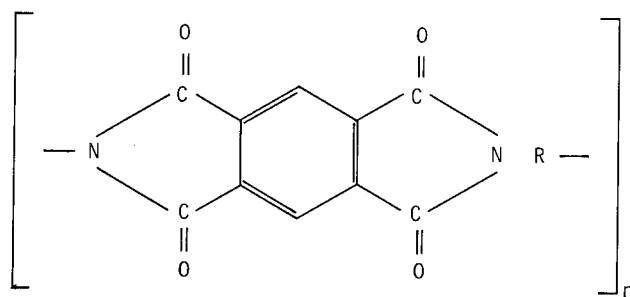


Figure 2. - Basic structure of aromatic polyimide. The structure consists of aromatic rings and heterocyclic molecules.

18 to 21). These polymers can be formed chemically by the reaction of pyromellitic dianhydrides and aromatic diamines.

Due to the multiple bonds between the aromatic and heterocyclic rings, the polyimides are characterized by a high thermal stability (400° C in air; 500° C in inert atmospheres) (refs. 19, and 22 to 24). They also have a high radiation stability (refs. 18, 19, and 22) and can withstand high exposure to neutrons, electrons, ultraviolet light, and gamma radiation. They are resistant to most common chemicals and solvents but are attacked by alkalis (refs. 19 and 22). At the decomposition point, they crumble to a fine powder without melting. For a more detailed discussion of the physical properties, see references 18 to 24.

## APPARATUS DESCRIPTION

The apparatus used to measure the friction coefficient and to evaluate wear lives of the solid lubricant films is illustrated in figure 1. Basically the device consists of a flat (6.3 cm diam) disk in sliding contact with a stationary (0.476 cm rad) hemispherically tipped rider. A 1-kilogram load is applied to the rider as the disk rotates at 1000 rpm. The rider slides on a 5-centimeter-diameter track on the disk, which gives it a linear sliding speed of 2.6 meters per second.

Induction heating is used to heat the disk. This is accomplished by placing an induction coil around the circumferential surface of the disk. The temperature is monitored by a thermocouple in contact with the disk surface. A micrometer is used to move the thermocouple away from the disk before it is set in motion. The temperature is then monitored by an infrared pyrometer which is focused on the wear track of the disk.

A strain gage senses the frictional force, which is continuously recorded on a strip-chart recorder.

## PROCEDURE

### Coating Formulation

The PI-bonded  $\text{MoS}_2$  films were prepared by mixing three parts (by weight) of  $\text{MoS}_2$  powder with one part (by weight) of polyimide solids. The PI-bonded  $(\text{CF}_{1.1})_n$  films were made by mixing three parts (by weight) of  $(\text{CF}_{1.1})_n$  powder with two parts (by weight) of polyimide solids. The density of  $(\text{CF}_{1.1})_n$  was only about one-half that of  $\text{MoS}_2$ ; therefore, the two film formulations contain equal volume percents of solid lubricant. A thinner consisting of N-methylpyrrolidone and Xylene was added to each formulation to obtain a sprayable solution.

### Disk Surface Preparation and Cleaning Procedure

The riders and disks were made of 440C stainless steel with a Rockwell hardness of C-60. In order to ensure good adherence of the solid film lubricants to the disks, they were roughened by sandblasting to an rms of  $0.90 \times 10^{-6}$  to  $1.25 \times 10^{-6}$  meter.

After the surfaces were roughened, they were scrubbed with a brush under running water to ensure that no abrasive particles remained. A water paste of levigated alumina was next rubbed gently over the surface with a clean polishing cloth. This was followed by a second scrubbing under running water. The disks were then rinsed in

distilled water and stored in a desiccator until they were coated with the solid lubricant.

The riders were first scrubbed with alcohol. Then a water paste of levigated alumina was applied with a polishing cloth. Cleaning continued until no trace of sediment from the rider appeared on the cloth. The riders were then rinsed in distilled water and stored in the desiccator until used.

## Application of Lubricant Films

Some tests were conducted using disks burnished with  $\text{MoS}_2$  or  $(\text{CF}_{1.1})_n$  powders. These coatings were applied by rubbing the powder onto the disk surface with the back of a napped polishing cloth. The polishing cloth was made of open weave fabric (twilled) and thus served as a good applicator.

The liquid mixture of polyimide and solid lubricant was sprayed onto each disk using an artist's airbrush. The coating did not dry rapidly. Thus, if more than a thin coat was applied, the liquid "ran" and a nonuniform coating resulted. To eliminate this "running" and to obtain the desired coating thickness, it was necessary to spray a thin coating of the formulation onto the disk, bake it at  $100^\circ\text{C}$  in an oven for 1 hour, and then spray another thin coating on the disk and repeat the procedure.

When the desired thickness of 10 to 20 micrometers was obtained, the remainder of the curing procedure was carried out. This procedure was to bake the coating at  $100^\circ\text{C}$  for 1 hour and then to bake it for an additional hour at  $300^\circ\text{C}$ . After the disks had cooled, some additional solid lubricant powder was rubbed onto the polyimide - solid lubricant film. This procedure was the same as that used for the burnished films.

## Test Procedure

The procedure for conducting the wear life tests was as follows: a rider and a disk (with the applied solid lubricant film) were inserted into the friction apparatus (fig. 1). The test chamber was sealed, and dry air (moisture content, 20 ppm) was purged through the chamber for 15 minutes. The flow rate was 1500 cubic centimeters per minute. This flow rate maintained a slight positive pressure in the chamber, which had a volume of 2000 cubic centimeters.

When the purge was completed, the temperature of the disk was slowly raised to the desired temperature by using induction heating. The temperature was held for 10 minutes to allow it to stabilize. The disk was then set into rotation at 1000 rpm, and a 1-kilogram load was applied.

The criterion for failure in these tests was a friction coefficient of 0.30. An auto-



matic cutoff system shut down the apparatus when the friction coefficient reached this value.

In order to calculate the rider wear rate at 25° C, the tests were stopped after 1 hour of sliding (60 kilocycles). The wear scar diameter on the hemispherically tipped rider was measured, and wear volume per hour was calculated.

For comparison, the wear rate and friction coefficient for unlubricated 440C stainless steel were also similarly determined. The same procedure was followed, but no cutoff friction coefficient was used.

## RESULTS AND DISCUSSION

### Friction Coefficient

The variation of the friction coefficient with time, at 25° C, in a dry-air atmosphere is shown in figure 3. The figure depicts actual friction traces for both the PI-

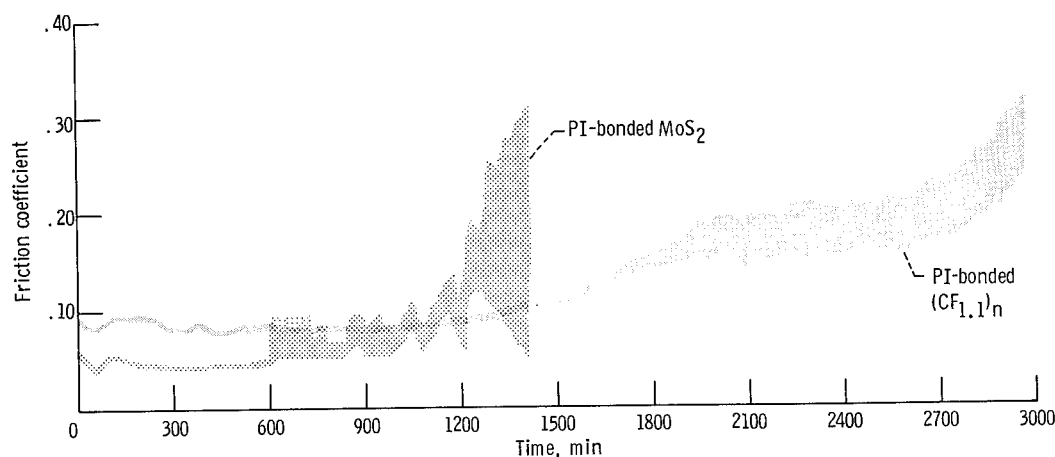


Figure 3. - Variation of friction coefficient with time for polyimide (PI)-bonded films of graphite fluoride ((CF<sub>1.1</sub>)<sub>n</sub>) and molybdenum disulfide (MoS<sub>2</sub>). Test temperature, 25° C; rider and disk material, 440C stainless steel; load, 1 kilogram; linear sliding speed, 2.6 meters per second; rotational speed, 1000 rpm; dry-air atmosphere (moisture content, 20 ppm).

bonded (CF<sub>1.1</sub>)<sub>n</sub> film and the PI-bonded MoS<sub>2</sub> film. The test conditions were a load of 1 kilogram, a sliding speed of 2.6 meters per second (1000 rpm), a dry-air atmosphere (moisture content, 20 ppm), and 440C stainless-steel riders and disks.

These friction traces are typical of all the tests conducted. Usually, at the start of each test, there was a run-in period (not shown in the figure) where the friction coefficient could attain a value as high as 0.20. This run-in lasted a few minutes, after which the friction coefficient fell to some minimum value. It then rose slightly and fluctuated around an equilibrium position. The remainder of the test consisted of a gradual in-

crease in the value of the friction coefficient until the cutoff friction coefficient of 0.30 was reached.

The friction coefficient of the PI-bonded  $(CF_{1.1})_n$  film seemed to be characterized by a more gradual increase with time than did the PI-bonded  $MoS_2$  film. The friction coefficient of the PI-bonded  $MoS_2$  film at some point during the test suddenly became erratic (e.g., at 600 min in fig. 3). This erratic friction is probably due to metallic contact through the lubricant film, and the subsequent rehealing of the film due to additional lubricant being brought into the contact region from the sides of the disk wear track.

The friction coefficients of PI-bonded  $(CF_{1.1})_n$  and PI-bonded  $MoS_2$  as a function of temperature are presented in figure 4. The values of the friction coefficient in this fig-

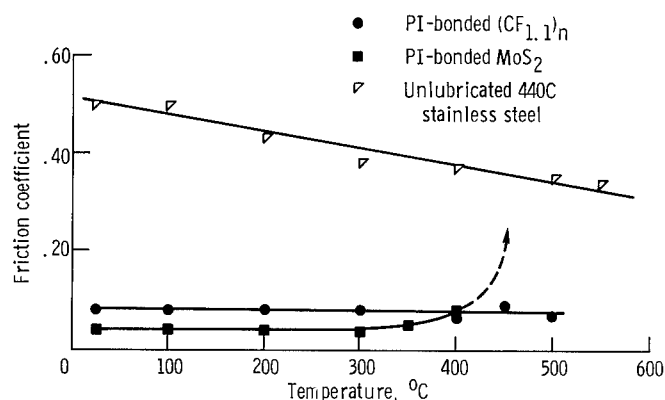


Figure 4. - Friction coefficient as function of temperature for PI-bonded graphite fluoride  $((CF_{1.1})_n)$  and PI-bonded molybdenum disulfide ( $MoS_2$ ) films applied to 440C stainless-steel disks. Rider material, 440C stainless steel; load, 1 kilogram; linear sliding speed, 2.6 meters per second; dry-air atmosphere (moisture content, 20 ppm).

ure and all following figures are the minimum values obtained for each test. Also shown in figure 4 are friction coefficient values for unlubricated 440C stainless steel sliding against itself. This figure illustrates that the friction coefficient of PI-bonded  $(CF_{1.1})_n$  remained constant at 0.08 over the entire test temperature range of 25° to 500° C. The friction coefficient of PI-bonded  $MoS_2$  remained constant at 0.04 over the range of 25° to 300° C. The friction coefficient increased to 0.05 at 350° C and to 0.08 at 400° C. At 450° C, the friction coefficient rose steadily, and no minimum value was recorded.

## Coating Wear Life

One of the most important properties that a good solid lubricant must possess is

the ability to remain intact on the surface to be lubricated for as long as possible. This quality, the wear life of the lubricant film, can be designated either as the time or the number of cycles until failure occurs.

Failure is an arbitrary term. It is usually the discretion of the experimenter which determines the onset of failure. In this study, the criterion for failure was a friction coefficient of 0.30. This value was less than the friction coefficient of unlubricated 440C stainless steel over the entire test temperature range.

The wear lives of four solid lubricant films were evaluated in this investigation. The films included rubbed-on (burnished) films of  $\text{MoS}_2$  or  $(\text{CF}_{1.1})_n$  and PI-bonded films of  $(\text{CF}_{1.1})_n$  and  $\text{MoS}_2$ . Figure 5 shows the wear life as a function of temperature for each of these four films. At each test temperature, the PI-bonded  $(\text{CF}_{1.1})_n$  films proved to be superior to the other three films.

The wear life of rubbed-on  $(\text{CF}_{1.1})_n$  was found to be at least four times the wear life of rubbed-on  $\text{MoS}_2$  over the entire test temperature range. PI-bonded  $(\text{CF}_{1.1})_n$  was found to be up to 10 times better than rubbed-on films of  $(\text{CF}_{1.1})_n$  over the same test temperature range. When the wear lives of the PI-bonded films were compared, it was found that, at  $25^\circ\text{C}$ , the wear life of the PI-bonded  $(\text{CF}_{1.1})_n$  was about twice that of the PI-bonded  $\text{MoS}_2$  film. As the temperature was increased, the relative difference

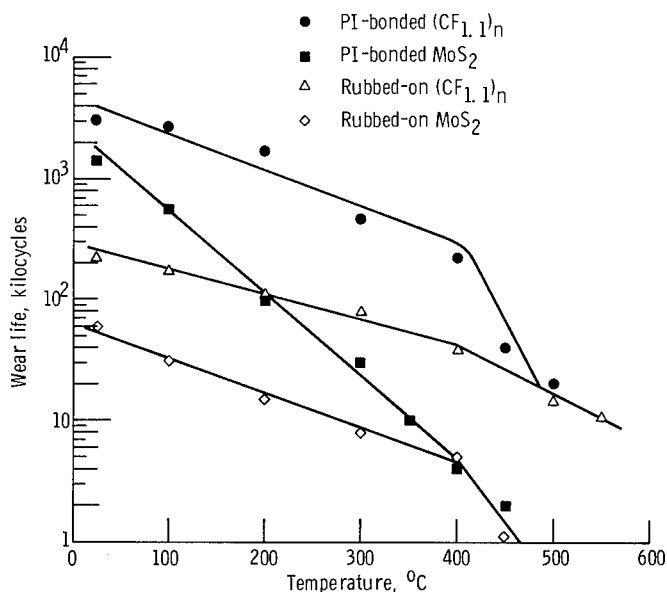


Figure 5. - Wear life as function of temperature for 440C stainless-steel disks lubricated with rubbed-on films and polyimide (PI)-bonded films of graphite fluoride ( $(\text{CF}_{1.1})_n$ ) and molybdenum disulfide ( $\text{MoS}_2$ ). Rider material, 440C stainless steel; load, 1 kilogram; linear sliding speed, 2.6 meters per second; dry-air atmosphere (moisture content, 20 ppm); failure criterion, a friction coefficient of 0.30.

between the wear lives became greater; and at 400° C, the difference was about a factor of 60.

At 450° C, the wear life of both PI-bonded films dropped off. Upon examination of the disks after test completion, it was found that the coatings had become powdery. Thus, the upper temperature limit for these PI-bonded films, in a dry-air atmosphere, is about 400° C. It is interesting to note that above 400° C the wear lives of the PI-bonded films approached the values of the wear lives of their respective rubbed-on solid lubricant films.

A comparison of the wear lives, at 25° C, for the four solid lubricant films is given in table I. The wear life of a commercially available sodium silicate bonded MoS<sub>2</sub> lu-

TABLE I. - COMPARISON OF WEAR LIVES

AT 25° C OF FIVE LUBRICANT FILMS

[Dry-air atmosphere (moisture content, 20 ppm); 440C stainless-steel riders and disks; 1-kg load; 2.6-m/sec linear sliding speed.]

Lubricant film	Wear life, kilocycles
PI-bonded (CF <sub>1.1</sub> ) <sub>n</sub>	2950
PI-bonded MoS <sub>2</sub>	1400
71-wt % MoS <sub>2</sub> - 7 wt % graphite - 22-wt % sodium silicate <sup>a</sup>	500
(CF <sub>1.1</sub> ) <sub>n</sub> rubbed film	230
MoS <sub>2</sub> rubbed film	60

<sup>a</sup>Commercially available lubricant tested by authors under above conditions.

bricant, which was tested under the same conditions, is also presented.

The rubbed-on films gave the shortest wear lives at 25° C. The wear life of rubbed MoS<sub>2</sub> was 60 kilocycles; rubbed-on (CF<sub>1.1</sub>)<sub>n</sub> films gave much better results, with a wear life of 230 kilocycles. The commercially available sodium silicate bonded MoS<sub>2</sub> film proved to be better than either of the rubbed-on films; its wear life was 500 kilocycles.

By using polyimide as the binder material for MoS<sub>2</sub>, further improvement in the wear life was achieved. The wear life for this film was 1400 kilocycles. When (CF<sub>1.1</sub>)<sub>n</sub> was substituted for MoS<sub>2</sub> in the polyimide film formulation, the results were even better. The wear life for these PI-bonded (CF<sub>1.1</sub>)<sub>n</sub> films was 2950 kilocycles.

It is important to note that no attempt was made to optimize the weight ratio of  $(CF_{1.1})_n$  to polyimide in formulating the film. An optimization study could result in an even better formulation.

The number of kilocycles at which the friction coefficient reached values of 0.10, 0.20, and 0.30 are given in table II. This information is given for each of the four lu-

TABLE II. - NUMBER OF REVOLUTIONS ELAPSED BEFORE FRICTION COEFFICIENT  
OF FOUR DIFFERENT FILMS REACHED VALUES OF 0.10, 0.20, AND 0.30

[Dry-air atmosphere (moisture content, 20 ppm); 440C stainless-steel disks and riders;  
1-kg load; 2.6-m/sec sliding speed. ]

(a) Test temperature, 25° C.

Friction coeffi- cient	PI-bonded ( $CF_{1.1}$ ) <sub>n</sub>	PI-bonded MoS <sub>2</sub>	Rubbed ( $CF_{1.1}$ ) <sub>n</sub>	Rubbed MoS <sub>2</sub>
	Number of revolutions, kilocycles			
0.10	1300	1000	20	57
.20	2250	1250	120	58
.30	2950	1400	230	60

(b) Test temperature, 100° C.

Friction coeffi- cient	PI-bonded ( $CF_{1.1}$ ) <sub>n</sub>	PI-bonded MoS <sub>2</sub>	Rubbed ( $CF_{1.1}$ ) <sub>n</sub>	Rubbed MoS <sub>2</sub>
	Number of revolutions, kilocycles			
0.10	2300	450	60	27
.20	2550	500	100	30
.30	2750	550	180	31

(c) Test temperature, 200° C.

0.10	1000	60	20	15
.20	1350	70	30	16
.30	1800	100	100	16

(d) Test temperature, 300° C.

0.10	350	20	10	3
.20	400	25	50	4
.30	450	30	80	8

(e) Test temperature, 400° C.

0.10	100	<1	20	2
.20	150	2	30	3
.30	230	4	40	5

(f) Test temperature, 500° C.

0.10	7	(a)	10	(a)
.20	10	(a)	14	(a)
.30	20	(a)	16	(a)

<sup>a</sup>Failed immediately.

bricant films and for each of the test temperatures of 25°, 100°, 200°, 300°, 400°, and 500° C. The wear-life ranking of the four films is the same at all three values of friction coefficient. Therefore, the choice of a friction coefficient of 0.10 or 0.20 instead of 0.30 as the failure criterion would not alter the conclusions concerning the relative durability of the coatings.

## Wear

In addition to providing a low friction coefficient and a long wear life, a good solid lubricant should also minimize wear. Thus, a series of experiments were performed to compare the wear occurring on the sliding riders.

Once a solid lubricant film fails, gross damage to the metal surface can take place. This damage is, of course, not characteristic of the wear prior to failure of the solid lubricant. The wear tests were thus stopped after 1 hour of sliding (60 kilocycles). The wear was determined by measuring the circular scar diameter on the hemispherically tipped rider and then calculating the wear volume of the material removed from the rider.

Figure 6 gives the results of tests which were run at 25° C with a 1-kilogram load and in a dry-air atmosphere (moisture content, 20 ppm). Four solid lubricant films

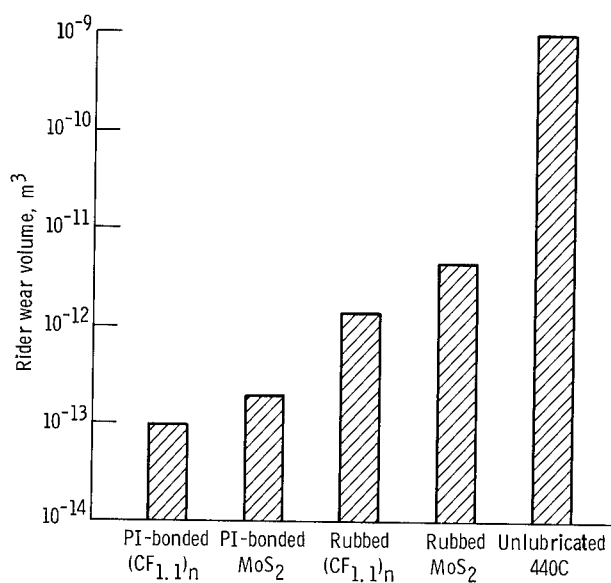


Figure 6. - Comparison of wear occurring to 440C stainless-steel riders after sliding for 1 hour against 440C stainless-steel disks coated with four different solid lubricant films. Test temperature, 25° C; load, 1 kilogram; linear sliding speed, 2.6 meters per second; dry-air atmosphere (moisture content, 20 ppm).

were tested: rubbed-on (CF<sub>1.1</sub>)<sub>n</sub>, rubbed-on MoS<sub>2</sub>, PI-bonded (CF<sub>1.1</sub>)<sub>n</sub>, or PI-bonded MoS<sub>2</sub>. The base metal, 440C stainless steel, was also run in the unlubricated condition.

The best results were obtained with the PI-bonded films. The wear occurring to the riders which slid on PI-bonded (CF<sub>1.1</sub>)<sub>n</sub> films was 1 × 10<sup>-13</sup> cubic meters, while the wear to the riders which slid on PI-bonded MoS<sub>2</sub> films was 2 × 10<sup>-13</sup> cubic meters. The wear occurring to the riders which slid on rubbed-on films was about 10 times greater than for riders which slid on PI-bonded films. The values were 1.5 × 10<sup>-12</sup> cubic meters for riders which slid on (CF<sub>1.1</sub>)<sub>n</sub> rubbed-on films and 4.0 × 10<sup>-12</sup> cubic meters for riders which slid on rubbed-on films of MoS<sub>2</sub>.

The wear of riders sliding on the unlubricated 440C stainless steel was  $1 \times 10^{-9}$  cubic meters per hour of sliding time (60 kilocycles); the PI-bonded films thus reduced the wear to 1/10 000th that of the unlubricated surface.

A photomicrographic comparison, at  $25^{\circ}\text{C}$ , of the wear occurring to the riders and disks is shown in figure 7. The disks were lubricated with PI-bonded  $\text{MoS}_2$ , or PI-bonded  $(\text{CF}_{1.1})_n$ . They were run in a dry-air atmosphere (moisture content, 20 ppm) until a friction coefficient of 0.30 was reached. This figure illustrates the fact that, even though the PI-bonded  $(\text{CF}_{1.1})_n$  film lasted over two times longer, the wear occurring to the riders and disks during their effective lives was nearly equivalent. The surface damage to the specimens was not particularly severe (fig. 7). Some metal-to-metal contact has probably taken place, but enough solid lubricant has found its way into the interface to provide protection.

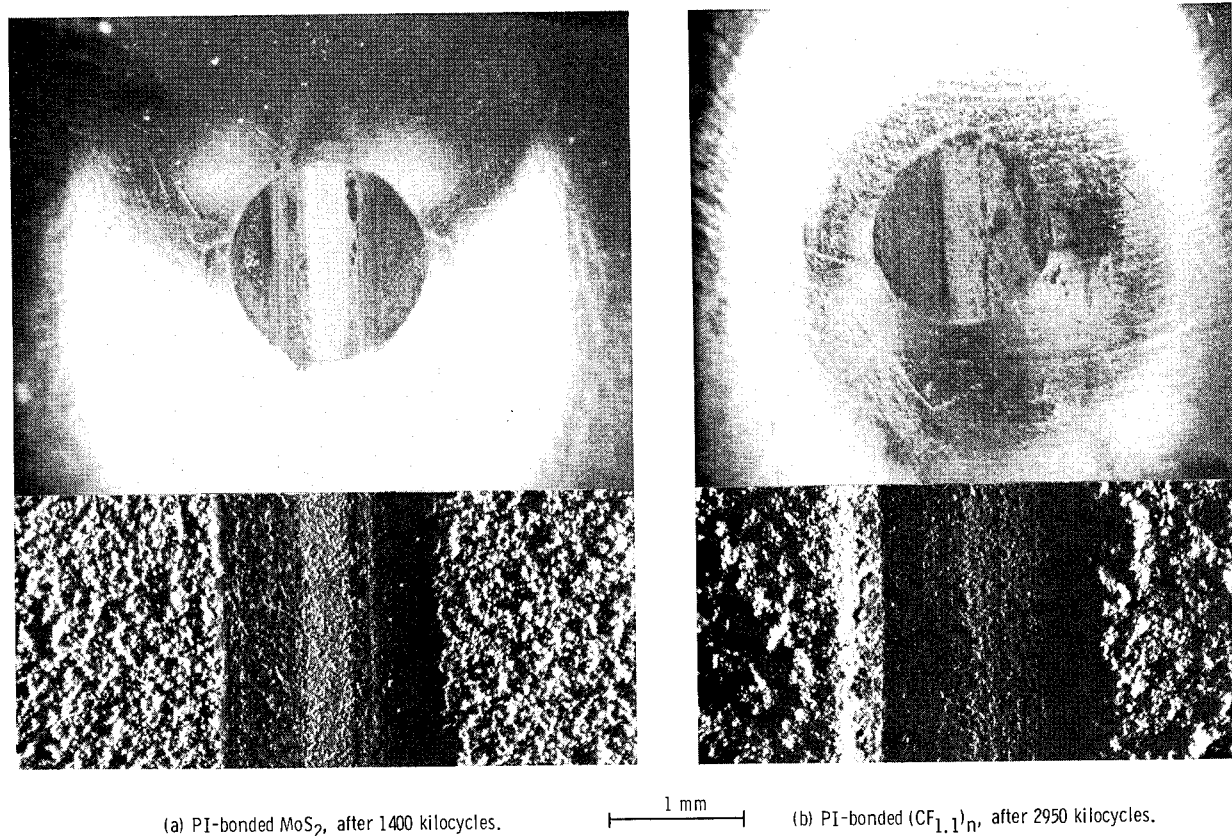


Figure 7. - Comparison of wear at  $25^{\circ}\text{C}$  of 440C stainless-steel riders and disks lubricated with coatings of polyimide (PI)-bonded molybdenum disulfide ( $\text{MoS}_2$ ) and PI-bonded graphite fluoride ( $(\text{CF}_{1.1})_n$ ). Linear sliding speed, 2.6 meters per second; load, 1 kilogram; dry-air atmosphere (moisture content, 20 ppm).

## SUMMARY OF RESULTS

A study was made of graphite fluoride as a solid lubricant in a polyimide binder. The results were as follows:

1. Polyimide (PI) is a suitable binder material for the solid lubricant graphite fluoride  $((CF_{1.1})_n)$ .
2. The wear life of PI-bonded  $(CF_{1.1})_n$  is superior to the wear life of similar films of PI-bonded molybdenum disulfide ( $MoS_2$ ).
3. The wear life of PI-bonded  $(CF_{1.1})_n$  is 10 times greater than the wear life of rubbed-on films of either  $(CF_{1.1})_n$  or  $MoS_2$ .
4. The PI-bonded  $(CF_{1.1})_n$  films performed exceptionally well at high temperatures; at  $400^\circ C$  for example, they lasted about 60 times longer than PI-bonded  $MoS_2$  films.
5. The rider wear rates of the PI-bonded  $(CF_{1.1})_n$  and PI-bonded  $MoS_2$  films were nearly equivalent at  $25^\circ C$ .
6. The reduction in the wear life of PI-bonded  $(CF_{1.1})_n$  above  $400^\circ C$  is due to the degradation of the polyimide.

Lewis Research Center,  
National Aeronautics and Space Administration,  
Cleveland, Ohio, December 13, 1971,  
132-15.

## REFERENCES

1. Fusaro, Robert L.; and Sliney, Harold E.: Preliminary Investigation of Graphite Fluoride  $(CF_x)_n$  as a Solid Lubricant. NASA TN D-5097, 1969. (Also Graphite Fluoride  $(CF_x)_n$  - A New Solid Lubricant. ASLE Trans., vol. 13, no. 1, Jan. 1970, pp. 56-65.)
2. E. I. du Pont de Nemours & Co.: Polyimides. British Patent 982, 914 (Concordance U.S. Patent 3,179,633), Feb. 10, 1965.
3. Buckley, D. H.; and Johnson, R. L.: Degradation of Polymeric Compositions in Vacuum to  $10^{-9}$  mm Hg in Evaporation and Sliding Friction Experiments. SPE Trans., vol. 4, no. 4, Oct. 1964, pp. 306-314.
4. Buckley, Donald H.: Friction and Wear Characteristics of Polyimide and Filled Polyimide Compositions in Vacuum ( $10^{-10}$  mm Hg). NASA TN D-3261, 1966.
5. Jones, William R., Jr.; Hady, William F.; and Johnson, Robert L.: Friction and Wear of Poly (Amide-Imide), Polyimide, and Pyrrone Polymers at  $260^\circ C$  ( $500^\circ F$ ) in Dry Air. NASA TN D-6353, 1971.



6. Loomis, William R.; Johnson, Robert L.; and Lee, John: High-Temperature Polyimide Hydraulic Actuator Rod Seals for Advanced Aircraft. NASA TM X-52889, 1970.
7. Campbell, M.; and Hopkins, V.: Development of Polyimide Bonded Solid Lubricants. Lub. Eng., vol. 23, no. 7, July 1967, pp. 288-294.
8. Schachner, Herbert: Carbon Monofluoride as Lubricant. German Patent 1,074,795 (Concordance British Patent 877122 and U. S. Patent 2,993,567), Feb. 4, 1960.
9. Ishikawa, T.; and Shimada, T.: Application of Polycarbon Monofluoride. Presented at the Fluorine Symposium, Moscow, 1969.
10. Gisser, H.; Petronio, M.; and Shapiro, A.: Graphite Fluoride as a Solid Lubricant. Proceedings of the International Conference on Solid Lubrication. ASLE SP-3, 1971.
11. Hawkins, W. L.: Thermal and Oxidative Degradation of Polymers. SPE Trans., vol. 4, no. 3, July 1964, pp. 187-192.
12. Mark, H. F.; and Atlas, S. H.: Principles of Polymer Stability. Polymer Eng. Sci., vol. 5, no. 3, July 1965, pp. 204-207.
13. Carothers, Wallace H.: Linear Polyamides Suitable for Spinning into Strong Pliable Fibers. U. S. Patent 2,130,523, Sept. 20, 1938.
14. Bower, G. M.; and Frost, L. W.: Aromatic Polyimides. J. Polymer Sci., Pt. A, vol. 1, no. 10, 1963, pp. 3135-3150.
15. Floyd, Don E.: Polyamide Resins. Second ed., Reinhold Pub. Co., 1966.
16. Edwards, Walter M.; and Robinson, Ivan M.: Polyimides of Pyrometallic Acid. U. S. Patent 2,710,853, June 14, 1955.
17. Edwards, Walter M.; and Endrey, Andrew L.: Polyimides. British Patent 903,271 (Concordance German Patent 1202981), Aug. 15, 1962.
18. Sroog, C. E.; Endrey, A. L.; Abramo, S. V.; Berr, C. E.; Edwards, W. M.; and Olivier, K. L.: Aromatic Polypyromellitimides from Aromatic Polyamic Acids. J. Polymer Sci., Pt. A, vol. 3, no. 4, 1965, pp. 1373-1390.
19. Heacock, J. F.; and Berr, C. E.: Polyimides - New High Temperature Polymers: H-Film, A Polypyromellitimide Film. SPE Trans., vol. 5, no. 2, Apr. 1965, pp. 105-110.
20. Adrova, N. A.; Bessonov, M. I.; Laius, L. A.; and Rudakov, A. P. (Kurt Ginkgold, trans.): Polyimides: A New Class of Thermally Stable Polymers. Technomic Publ. Co., 1970.

21. Devine, M. J.; and Kroll, A. E.: Aromatic Polyimide Compositions for Solid Lubrication. *Lub. Eng.*, vol. 20, no. 6, June 1964, pp. 225-230.
22. Todd, N. W.; and Wolff, F. A.: Polyimide Plastics Withstand High Temperatures. *Materials in Des. Eng.*, vol. 60, no. 2, Aug. 1964, pp. 86-91.
23. Bower, G. Ronald: What Engineers Should Know about Self-Lubricating Plastics. *Metal Prog.*, vol. 88, no. 3, Sept. 1965, pp. 106, 108, 110, 112, 114.
24. Freeman, James H.; Frost, Lawrence W.; Bower, George M.; and Traynor, Edward J.: Resins and Reinforced Plastic Laminates for Continuous Use at 650° F. *SPE Trans.*, vol. 5, no. 2, Apr. 1965, pp. 75-83.

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION  
WASHINGTON, D.C. 20546

OFFICIAL BUSINESS  
PENALTY FOR PRIVATE USE \$300

FIRST CLASS MAIL

POSTAGE AND FEES PAID  
NATIONAL AERONAUTICS AND  
SPACE ADMINISTRATION



014 001 C1 U 15 720218 S00942DS  
DEPT OF THE ARMY  
PICATINNY ARSENAL  
PLASTICS TECHNICAL EVALUATION CENTER  
ATTN: SMUPA-VP3  
DOVER NJ 07801

3401

POSTMASTER: If Undeliverable (Section 158  
Postal Manual) Do Not Return

*"The aeronautical and space activities of the United States shall be conducted so as to contribute . . . to the expansion of human knowledge of phenomena in the atmosphere and space. The Administration shall provide for the widest practicable and appropriate dissemination of information concerning its activities and the results thereof."*

—NATIONAL AERONAUTICS AND SPACE ACT OF 1958

## NASA SCIENTIFIC AND TECHNICAL PUBLICATIONS

**TECHNICAL REPORTS:** Scientific and technical information considered important, complete, and a lasting contribution to existing knowledge.

**TECHNICAL NOTES:** Information less broad in scope but nevertheless of importance as a contribution to existing knowledge.

**TECHNICAL MEMORANDUMS:** Information receiving limited distribution because of preliminary data, security classification, or other reasons.

**CONTRACTOR REPORTS:** Scientific and technical information generated under a NASA contract or grant and considered an important contribution to existing knowledge.

**TECHNICAL TRANSLATIONS:** Information published in a foreign language considered to merit NASA distribution in English.

**SPECIAL PUBLICATIONS:** Information derived from or of value to NASA activities. Publications include conference proceedings, monographs, data compilations, handbooks, sourcebooks, and special bibliographies.

**TECHNOLOGY UTILIZATION PUBLICATIONS:** Information on technology used by NASA that may be of particular interest in commercial and other non-aerospace applications. Publications include Tech Briefs, Technology Utilization Reports and Technology Surveys.

*Details on the availability of these publications may be obtained from:*

**SCIENTIFIC AND TECHNICAL INFORMATION OFFICE**

**NATIONAL AERONAUTICS AND SPACE ADMINISTRATION**

**Washington, D.C. 20546**